

Review Assignment 1

Dislocation Structures in Nickel Microcrystals

Joy Perkinson, 10/06/08

The behavior of dislocation structures in micro- and nanoscale crystals has not been thoroughly studied, and is not yet well-understood. The article “Dislocation structures and their relationship to strength in deformed nickel microcrystals” quantitatively investigates the dislocation behavior and strengthening of pure nickel microcrystals under uniaxial compression. The results exhibit increased microcrystal strength and dislocation density at smaller microcrystal size. A variety of theories have been suggested to explain these results, and the article refines and further researches these theories.

The microcrystals used in the study were nickel cylinders with diameters ranging from 1 to 20 μm with height-to-diameter aspect ratios of 2.5 to 1. The cylinders started with an initial dislocation density on the order of $\rho_o = 10^{13}\text{m}^{-2}$. The cylinders were loaded in uniaxial compression along the [269] direction. Strain rates of 10^{-4}s^{-1} were achieved through increasing load and load hold. After deformation, TEM films were extracted from the microcrystals through focused ion beam (FIB) milling. Most films in the study were taken from the primary slip system, $(\bar{1}11)[101]$, which is typical of nickel’s FCC crystal structure. However, three slip systems were found to be active within the smaller microcrystals. Dislocation density was primarily measured via the “line-intercept” method, in which five lines were randomly drawn through the TEM images and the intersection of dislocation lines with drawn lines were counted. Dislocation density was approximated by the number of intersection points divided by the product of the line lengths and the foil thickness. The initial dislocation density, ρ_o , was found to be $1.4 \times 10^{14}\text{m}^{-2}$.

Much of the discussion in the article discussed the “mean-field reference stress” (MFRS), which is a combined effect due to lattice friction, initial dislocation forest density, and the stress required to activate dislocation sources. It was hypothesized that an “effective stress” must exceed the MFRS components in order for dislocation evolution to occur. However, even when the MFRS model was corrected for the larger dislocation density in smaller microcrystalline samples, the magnitude of the calculated MFRS was not large enough to account for the large flow stresses sustained in small sample sizes prior to unloading. The “exhaustion hardening” theory suggests that there

may be an altered forest-hardening response at small scale lengths that approach the dislocation length. At this characteristic length, ξ^* , the weakest slip planes are removed from the volume via a statistical effect. However, the only description of ξ^* provided so far applies only to macroscale crystals in Stage II glide, not microcrystals in Stage I glide. Overall, the article raised more questions than it answered, and dislocation behavior in microcrystals is still not well-understood. However, the article suggested many possible avenues of further research.

This paper complemented the 3.14 course material very well. Though dislocation mechanisms in microcrystals are not well-understood, the discussion of dislocation forests, “dislocation braids,” and the way they interact with slip plane boundaries, the crystal edges, and pinning points helped develop a more intuitive understanding of dislocation interactions. Additionally, the images of dislocations exhibiting directional preference in the $(\bar{1}11)$ slip plane visually demonstrates the workings of slip systems.

While the article helped provide an overall intuitive understanding of dislocation structures in microcrystals, the sources of error were not handled as thoroughly as they could have been. Some sources of error were taken into account to create scale bars, such as the $\pm 5\%$ error due to TEM film thickness variation and the 30% underestimation in dislocation line-intersections due to difficulties inherent in imaging dislocations that are close together. However, other sources of error, such as the 10% error due to using the line-intercept method of dislocation density measurement, were mentioned briefly but then neglected. Though the data strongly suggest trends, the article would be helped by a more thorough analysis of the error that includes a worst-case-scenario error calculation, especially given the amount of variation between measured values of dislocation density and flow stresses (e.g. Fig 9, 13, and 14).

There are a few simple experiments that could investigate some of the uncertainty in the paper. For example, section 4.1 reports that some papers have suggested that imaging forces deplete samples of dislocations. This could be tested by imaging the same TEM foil multiple times and carefully measuring the dislocation density over time. If dislocations did start disappearing, then the rate of depletion could be quantized and taken into account as a source of error. Another source of uncertainty in the article was the purely qualitative comparison between the dislocation structures in the microcrystals versus bulk nickel. The article mentions the chance that the microcrystals were fabricated from a high-dislocation-density region of the bulk

material, leading to a higher dislocation density. Given that the main result being reported on is the unusually high strength of microcrystals, it would make sense to take microcrystals from a variety of different areas within the bulk material to ensure that the microcrystals were not more work-hardened than expected. This simple experiment would help cement the validity of the size-dependent strength results.

One useful but time-consuming area of further research could be running large-scale simulations. One would need access to a fast computer to make simulations feasible, but if such a computer were available, simulations could lead to a better understanding of the strengthening mechanisms and dislocation behavior in microcrystals. Simulations would provide a more comprehensive picture of the dislocation structures throughout the entire microcrystal, as opposed to on a single plane at a time. Furthermore, the theory that the average dislocation density is stored during early stages of plastic deformation, as hypothesized in section 5, could be observed.

While simulations would be a useful area of further research, however, the clearest area of further research is in generalizing the conclusions of this article. Different crystal materials, crystal structures, sample sizes, aspect ratios, geometries, deformation techniques, and initial dislocation densities should be investigated. For example, in order to study and characterize the correlation length ξ^* , values of ξ^* could be determined for a wide range of samples and then compared to various physical properties of the materials. Such an approach could suggest what ξ^* is related to, and could thus help describe the correlation length. A similar approach could help determine the nature of the mechanism that causes the observed flow stresses to exceed MFRS predictions.

Another way to generalize the conclusions of the article would be to conduct a more exhaustive study on the way a broad range of microcrystal sizes affects the dislocation structures and sample strength. The discrepancy between MFRS predictions and the observed effective stresses prior to unloading appears fairly constant in the range studied in this paper (see Fig. 14), but the mechanisms causing the discrepancy are unknown. Examining how this discrepancy changes at smaller and larger sample diameters could provide insight into its cause. For example, given that this discrepancy is characteristic of microcrystals, one could research sample diameters greater than $20\mu\text{m}$ to find the point at which the discrepancy is no longer observed. A broad study of smaller microcrystals around $1\mu\text{m}$ and less could also be useful in explaining the unique stress-strain behavior of the $1\mu\text{m}$ microcrystal

observed in Figure 4b.

Overall, this article provides insight into the questions facing the still-developing field of dislocation theory, and complements the discussion of dislocations presented in 3.14. The results of the paper are not well-generalized, though, and much further research is needed. However, further research will hopefully lead to important new advances in the understanding of microcrystal behavior, which will become increasingly important as we continue into the era of nanoscale devices.